भारतीय मानक
Indian Standard

IS 12869 (Part 1) : 2022

कांच की श्यानता और श्यानतामितीय बिंदु ज्ञात करने की पद्धति

भाग 1 परीक्षण की मानक पद्धतियाँ

(पहला पुनरीक्षण)

Methods for Determination of Viscosity and Viscometric Fixed Points of Glass

Part 1 Standard Test Methods

(First Revision)

ICS 81.040.01

© BIS 2022

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

June 2022 **Price Group 8**

Glass, Glassware and Laboratoryware Sectional Committee, CHD 10

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Glass, Glassware and Laboratoryware Sectional Committee had been approved by the Chemical Division Council.

The quality and the type of glass is very much dependent upon the relationship of viscosity of glass with temperature change. Important information of glass technology regarding the temperature required to produce a homogeneous melt for production of glass and the temperature range of formation of glass can be collected from this relationship. This standard, therefore, also provides guidance for determining viscosity/temperature relationship.

This standard was originally published in 1989 based on ISO 7884-3 : 1987 'Glass — Viscosity and viscometric fixed points — Part 3 : Determination of viscosity by fibre elongation viscometer'. However, to keep it in line with the improved technology, the first revision of this standard has been undertaken. In this first revision, two new methods for determination of viscosity of glass have been added. The parallel plate viscometer method is based on ASTM C 1351M – 96 (2017) 'Standard Test Method for Measurement of Viscosity of Glass between 10^4 Pa.s and 10^8 Pa.s by Viscous Compression of a Solid Right Cylinder [Metric]' and the Beam Bending Viscometer method is based on ASTM C 1350M – 96 (2019) 'Standard Test Method for Measurement of Viscosity of Glass between Softening Point and Annealing Range (Approximately 10^8 Pa.s to Approximately 10^{13} Pa.s) by Beam Bending [Metric]'

This standard forms Part 1 of Methods for determination of viscosity and viscometric fixed points of glass. Part 2 of this standard deals with determination of softening points and Part 3 deals with determination of annealing and strain point by beam bending method.

The departure from ISO document is given at Annex E.

The composition of the Committee responsible for formulation of this standard is given in Annex F.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

Indian Standard

METHODS FOR DETERMINATION OF VISCOSITY AND VISCOMETRIC FIXED POINTS OF GLASS

PART 1 STANDARD TEST METHODS

(First Revision)

1 SCOPE

This standard (Part 1) prescribes the methods for determining the viscosity of glass. This standard includes following three methods:

a) Fibre elongation method;
b) Beam bending viscometer method; and
c) Viscous compression of a solid right cylinder method.

2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

IS No.	*Title*
7358 : 1984	Specification for thermocouples (*first revision*)

3 TERMINOLOGY

3.1 Viscosity — It is defined as the proportion of the shear stress which is applied and the shear rate that is being generated. In case of a Newtonian liquid this connection is determined by the relation given as:

$$\text{Shear stress} = \text{Viscosity} \times \text{Shear rate}$$

$$[\mathrm{N/m^2}] = [\mathrm{Ns/m^2}].[1/\mathrm{s}]$$

The unit of viscosity is Ns/m^2 or Pa.s (Pa = N/m^2), in accordance with the Standard International System. The unit Poise was previously used. As the glass industry is still frequently using the unit Poise, it is imperative to give a correct description of the difference in between Pa.s and Poise.

NOTE — In accordance with the materials science, a material is said to be "Newtonian" in case it exhibits a linear relationship between strain rate and stress.

$$1\ dPa.s = \frac{dNs}{m^2} = 1P,\ 1\ dPa.s = 0.1\ Pa.s,\ 1\ Pa.s = 10\ P.$$

where dPa.s is deci pascal second, $\frac{dNs}{m^2}$ *is decinewton second per square metre and P is poise.*

3.2 Range of Viscosity

In regard to the practical application, three different ranges of viscosity measurement can be distinguished as:

a) *annealing range* — from 10^{13} to 10^{15} dPa.s;
b) *melting range* — up to 10^3 dPa.s; and
c) *working range* — from 10^3 to 10^8 dPa.s.

3.3 Viscometric Fixed Points

It is quite convenient to give preference to the following five temperatures in order to characterize the viscosity/temperature behaviour of a glass.

NOTE — The expression 'fixed point' does not denote any relation to thermodynamical fixed points.

3.3.1 *Working Point* T_{10}

The temperature that corresponds to a viscosity (η_{10}) of 10^4 dPa.s.

3.3.2 *Liquidus Point* T_{11}

The liquidus temperature T_{11} is the temperature above the point where no crystallization is possible. This is the maximum temperature at which crystals can co-exist with the melt. This liquidus temperature corresponds to the viscosity (η_{11}) $10^{4.8}$ dPa.s. However, the real liquidus temperature also depends on the composition (and the contaminants present) in the glass and is determined by the thermodynamics that can be derived from phase diagrams.

3.3.3 *Softening Point* T_{12}

It is the temperature above that the glass is capable of most of the forming operations. The softening point is also known as Littleton temperature. The corresponding viscosity (η_{12}) is estimated by the following equation:

$$\eta_{12} = 2.1 \times 10^7 (\rho - \sigma/520)$$

where

ρ = density of glass, in grams per cubic centimetre; and

σ = surface tension, in millinewton per metre.

For ρ = 2.5 g/cm^3 and σ = 300 mN/m, the viscosity is

$$\eta_{12} = 10^{7.6} \text{ dPa.s}$$

3.3.4 *Annealing Point* T_{13}

It is the temperature at which internal stresses that are present in a glass are substantially relieved in just a matter of minutes. At this temperature the annealing point corresponds to a viscosity (η_{13}) from 10^{13} to $10^{13.2}$ dPa.s.

3.3.4.1 *Annealing range*

It is the temperature range in which stresses present in glass articles can be relieved at a commercially desirable rate.

3.3.5 *Strain Point* T_{14}

It is the temperature at which internal stresses present within a glass are substantially relieved in just a matter of hours. Temperature at the strain point corresponds to a viscosity (η_{14}) from $10^{14.5}$ to $10^{14.7}$ dPa.s.

3.4 Fibre

It is the cylindrical part of the specimen bearing a constant cross-section S = $\frac{\pi d^2}{4}$ along its entire length where *d* is the uniform diameter of the fibre which is drawn from the molten glass.

3.5 Fibre Ball

A fibre that has two of its ends in the shape of spherical balls and is prepared carefully by fusing each end of the fibre on the burner flame into spherical ball shape enabling the suspension of the fibre vertically, the upper ball resting on a suitable slot in a fixed support while a loading devise with a suitable slot hangs supported on the lower ball.

3.6 Load

It comprises of all the parts of an apparatus on which gravity can act to produce a force on the fiber's lower end.

3.7 Loading Pieces

It is a device for suspending the loading pieces (corresponding to a range of loads) below the specimen in an axial direction including the lower extension rod.

3.8 Dead Weight

It stems from the part of the fibre which is below the cross-section being under consideration. There is a linear variation for the force due to gravity from zero at the lower end to the upper end of the fibre.

3.9 Surface Tension Effect

This effect acts along the length of fibre thereby shortening the length. It thus, opposes the gravitational force because of the load.

3.10 Beam Bending Viscometer

It is a device that is used to find the viscosity of a glass from 10^8 Pa.s to 10^{13} Pa.s approximately by measuring the deflection rate of a simply supported beam. The equation for calculating viscosity by this method is:

$$\eta = \frac{gL^3}{1440\, I_c \left(dh/dt\right)}\left[M + \frac{\rho AL}{1.6}\right]\left[\frac{\left(1+\alpha_s T\right)^3}{\left(1+\alpha_g T\right)^4}\right]$$

where

M = load (applied load + loading train), gm;

η = viscosity, Pa.s;

g = acceleration of gravity, 980 cm/s^2;

dh/dt = midpoint deflection rate of test beam, cm/s;

ρ = density of glass, g/cm^3;

IC = cross-sectional moment of inertia, cm^4;

A = cross-sectional area of the beam, cm^2;

L = support span, cm; and

αs and αg = mean coefficient of linear thermal expansion of support stand and glass, respectively, 25 °C to temperature of measurement, T, m/m/°C. *See* Note below.

NOTE — The term $(1 + \alpha_s T)^3$ / $(1 + \alpha_g T)^4$ corrects for thermal expansion changes of room temperature dimensions. It can be ignored when α_s and α_g are approximately equal. A fused silica support stand in combination with a high expansion glass can make this term 3 percent in magnitude. Only an estimate of α_g is required, since the correction is small. Use 1.5 times the room temperature coefficient if data are unavailable.

3.11 Parallel Plate Viscometer

It is a device which is used to determine the viscosity of glass between 10^4 Pa.s to 10^8 Pa.s approximately by the measurement of the rate of change in thickness of a cylindrical shaped specimen between parallel plates which is moving perpendicular to the common central axis. For calculating the viscosity by the parallel plate method, following equation can be used:

$$\eta = 2\pi \frac{Mgh^5}{30V(dh/dt)(2\pi h^3 + V)(1+\alpha T)}$$

where

η = viscosity, Pa.s;
M = applied load, g;
g = acceleration due to gravity, 980 cm/s^2;
t = time, s;
V = specimen volume, cm^3;
h = specimen thickness at time t, cm;
dh/dt = compression rate, cm/s; and
α = glass mean coefficient of thermal expansion, 25 °C to the measurement temperature, T, m/m/°C. *See* Note below.

NOTE — The term ($1 + \alpha T$) corrects for the specimen dimensional changes due to thermal expansion. For low thermal expansion glasses, it can be ignored. However, for a glass with an α of 20×10^{-6} / °C at a measurement temperature of 1 000 °C, this term produces a correction of 2 percent. Only an estimate of α is necessary since the correction is small. Use twice the room temperature coefficient if data are unavailable.

ANNEX A

(*Clause* 1)

FIBRE ELONGATION METHOD

A-1 OUTLINE

This method prescribes the method for determining the viscosity of glass by (fibre elongation method, measuring the elongation of a glass fibre under a defined uniaxial stress. In addition, the viscosity/temperature relationship (*see* Annex D) and the dependence of viscosity on thermal history can be determined.

NOTE — The viscous flow by the glass fibre is assumed to be Newtonian. The glass is presumed to be homogeneous and isotropic and the effect of gravity on fibre is neglected. When the fibre shape and cross-sectional tolerance conform to sample specifications given in this standard, all parts of the fibre can be presumed to be subjected to the same mechanical stress leading to a pure elongation.

A-1.1 The knowledge of equilibrium viscosities in the low temperature range with their dependence on temperature and thermal history will be extremely useful in predicting glass properties and behavior.

A-2 FIELD OF APPLICATLON

A-2.1 The methods are applicable mainly to glass viscosity range from 10^8 to 10^{18} dPa.s with possibilities for extending the range to $10^{14.5}$ dPa.s with special devices. This corresponds to a temperature range of 800 °C to 400 °C for normal commercial glass compositions being produced in bulk quantities.

NOTE — In the viscosity ranges above 10^{12} dPa.s the thermal equilibrium of the glass structure takes considerable time to readjust following a temperature change. If equilibrium measurements are envisaged, sufficient time must be allowed following the change of temperature for thermal equilibrium to be re-established before the elongation measurement is undertaken. Alternatively a dynamic measurement can be done involving measurements made during a well-defined time/temperature programme. These measurements will correspond to a dynamical, non-equilibrium value for the viscosity η_{neq} indicated by which may nevertheless be consistent and reproducible under defined time temperature programme. The symbol without index denotes always equilibrium viscosity.

A-3 PRINCIPLE

A-3.1 Basic Relation

Consider the force F_O of the load acting on a specimen prepared from a glass with Newtonian flow behaviour and with no defects. With surface tension and the dead weight of the fibre being neglected, equation (1) gives a good approximation for the extensional flow rate $\frac{dl}{dt}$ at constant volume V.

$$\frac{dl}{dt} = \frac{1}{3} \times \frac{1}{\eta} \times \frac{F_o . l^2}{V} \qquad \text{... (1)}$$

where

η = viscosity, in decipascal second (dPa.s);

V = volume of the fibre;

$$F_O = mg$$

where

m = total mass contributing to F_o:

g = acceleration due to gravity; and

l = length of the fibre.

From the measurement of length as a function of time for a given load, a straight line plot of $\frac{dl}{dt}$ versus l^2, can be drawn.

From the slope of the straight line η can be calculated. In practice, the initial length l_o of the fibre is estimated independently and the elongation (increases in length) is measured by observing a fixed mark on the loading device by using a travelling microscope/cathetometer provided with a vernier scale or an eye piece micrometer.

A-3.2 Order of Magnitudes of Elongation

For an elongation from l_1 to l_2; ($l_2 - l_1 = \delta l$) during the time Δt an expression is obtained by integrating and rewriting (1) as:

$$\eta = \frac{g}{3} \times m \frac{\Delta t}{V} \times \frac{l_2 \times l_1}{l_2 \times l_1} \qquad \text{... (2)}$$

$$32\ 700 \times m \times \Delta t \times \frac{l_1}{V} \times \frac{(l + \delta l / l_1)}{\delta l / l_1} \qquad \text{... (3)}$$

Where, l_1, l_2, δl; are in millimetre, V is in cubic millimeters, m is in g, Δt is in seconds and η is in dPa.s. For a given diameter of the fibre at the start of experiment d (presuming a constant volume $V = \frac{\pi d^2}{4} \times l_l$) it is possible to calculate the time interval for extension ratio $\frac{\delta l}{l_1}$ of say 0.02 at different loads for a given viscosity. These values of t_s are tabulated (*see* Table 1) for an initial diameter of the fibre at d = 0.737 mm and $\frac{\delta l}{l_1} = 0.02$.

From the Table 1, it is clear that for the same extension ratio, for a range of temperatures for which the viscosity increases by six orders of magnitude (10^8 to 10^{14}) the extension ratios have to be correspondingly reduced by almost similar orders of magnitude in order to keep reasonably convenient time intervals. This implies increasing the accuracy of measurement of δl by similar orders of magnitude.

Table 1 Time Interval t_s for an Extension $\frac{\delta l}{l_1}$ = 0.02 under a Load of Mass *m*

(As Corrected for Dead Weight and Surface Tension), for a Fibre of Initial Diameter d_1 = 0.737 mm

(*Clauses* A-3.2, A-4.4.2)

Sl No.	Viscosity *dPa.s*	Time Interval t_s for in Seconds for Load *m*				
		1 g	4 g	16 g	64 g	256 g
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	10^8	25.6	–	–	–	–
ii)	10^9	256	64	–	–	–
iii)	10^{10}	2 560	640	160	40	–
iv)	10^{11}	25 600	6 400	1 600	400	100
v)	10^{12}	256 000	64 000	16 000	4 000	1 000
vi)	10^{13}	–	–	160 000	40 000	10 000
vii)	10^{14}	–	–	–	–	100 000

A-4 APPARATUS

A-4.1 Viscometer Furnace

A vertical mounted muffle furnace with both ends open, being electrically heated and with a temperature range of up to 900°C is suitable. It must have provisions for measuring the temperature and its distribution along the length of the fibre specimen using thermo-couples. The temperature gradient in the region of the specimen shall not exceed 0.2°C/cm.

A-4.1.1 The temperature control must be effected so as to ensure a consistency of temperature in respect of time at any point within the working space to within ± 1°C or better. Temperature variations must be effected with consistent and repeatable linear programmes.

A-4.2 Temperature Measuring and Indicating Instruments

A-4.2.1 Alumina insulated platinum — 10 percent rhodium/platinum (Type S of IS 7358 : 1984) thermocouples or nickel-chromium/nickel (Type K of IS 7358 : 1984) thermocouples shall be employed. They shall have low thermal inertia with wire diameters not more than 0.5 mm and sufficient immersion length of the hot junction inside the space where temperature is measured.

A-4.2.2 Control thermocouple hot junctions should be located as close as possible to the furnace heating elements to ensure fast response between any temperature variation and the corrective action by the controls. The hot junctions of the thermocouples for measuring specimen temperatures should be close to specimens (*see* Fig. 1). The temperature distribution along the length of the fibre shall be monitored by suitable devices, for example:

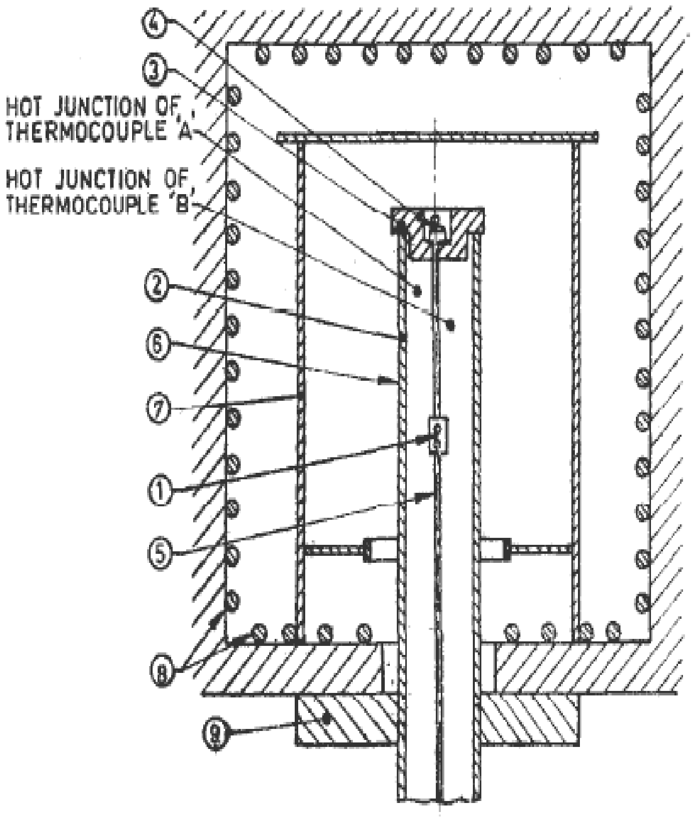
a) a nickel block of high thermal mass surrounding the fibre in a cylindrical shape with a number of holes positioned at various points distributed evenly covering the length of the fibre thermocouples being inserted through the holes, and
b) Alternatively, a mobile thermocouple of low thermal mass (*see* Fig. 1).

A-4.2.2.1 The thermocouples shall be calibrated in accordance with the procedure given in IS 7358 : 1984.

A-4.2.3 The electrical outputs of the thermocouples shall be measured potentiometrically, drawing nil currents or by a high resistance electronic amplifiers having a sensitivity of 1 microvolt for Type *S* (IS 7358 : 1984) and of 4 microvolt for Type *K* (IS 7358 : 1984) thermocouples. The cold junctions of the thermocouples shall be maintained at 0°C throughout; alternatively the measuring instrument shall be provided with automatic cold junction compensation device.

A-4.3 Hanging Device for Specimen and Load

The fibre is freely suspended from a connecting device by placing the upper ball of fibre in the lower groove of the device (Fig. 2 and Fig. 3). A fused silica rod with ball ends is used as an upper extension for the suspension, the lower ball of this rod supports the connecting device at its upper groove. The upper ball of the silica rod rests centrally on a suitable groove of metal plate of ceramic plate placed on the top of the muffle tube. The loading linkage consists of a fused silica rod of about 1 mm diameter with fused balls on both ends. The upper ball rests on the lower groove of the connector which is suspended from the fibre. A similar connector hanging from the lower end of this rod and extending outside the furnace, can be used for loading.


4
3
HOT JUNCTION OF THERMOCOUPLE 'A'
HOT JUNCTION OF THERMOCOUPLE 'B'
2
6
7
1
5
8
9

1 Connector for the lower ball of the test specimen and the and the loading device (for connectors offering different magnitudes of load, *see* Fig. 3)

2 Test specimen

3 Support plate

4 Upper ball of the specimen

5 Loading device (only the road is shown)

6 Support-stand : tube of fused silica

7 Screening between working space and heaters

8 Chamber furnace with heaters

9 Cover ring below the furnace (fixed at the silica tube)

Fig. 1 Fibre Elongation Method: Example of A Testing Device (Schematic)

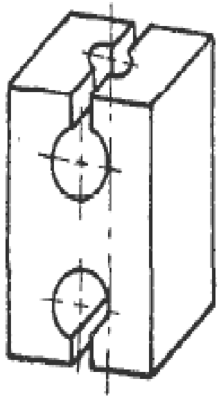


Fig. 2 Connector Block (Metal) Bored To Act As A Hanging Device

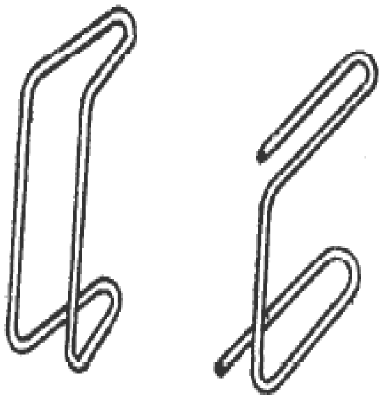


Fig. 3 Connector Hooks (Wire) To Act As A Hanging Device

A-4.3.1 It is absolutely essential that the extension rods and the fibre hang vertically, coaxially and centrally with reference to the furnace muffle tube. While measuring viscosities in the range of 10^8 to 10^9 dPa.s, the loading may entirely consist of only the lower extension rod of fused silica of approximately 0.5 mm diameter and a total mass of about 1 g. No additional loading is necessary.

A-4.4 Loading Pieces

While measuring medium viscosity ranges 10^{10} to 10^{11}, the total load on fibre should be around 4 g. The lower silica extension rod should have a diameter around 1 mm.

A-4.4.1 For higher viscosities heavier loads will be necessary and the lower silica extension rod must have a larger diameter.

A-4.4.2 A set of brass loading pieces with masses determined to an accuracy of 0.01 g may be used. The pieces shall be shaped suitably, to be hung symmetrically from a hook which can be connected to the lower extension fused silica rod by the connecting device. An idea of the masses for the brass pieces can be had from Table 1.

A-4.5 Measurement of Elongation

A-4.5.1 *Use of Optical Devices*

A mark is made on the load carrying device outside the furnace. A cathetometer or travelling microscope fitted with scale and vernier can be used. The vernier must have a least count of at least 0.02 mm. The error arising from graduation of the scale and vernier shall be such as to ensure that the error in measurement of δl does not exceed 0.01 mm. The instrument (travelling microscope or cathetometer) shall be rigidly locked in position during any one series of determinations. It shall be conveniently positioned in advance so that the maximum extension of the fibre will still be within the range of its observation.

A-4.5.2 *Measurement of the Initial Length l_1*

The length l_0 of the fibre between the two ball shaped ends is measured at the room temperature using a travelling microscope or cathetometer. The fibre is then suspended in position with the extension rods, connectors and loading device in the furnace at room temperature. The measuring instrument is then focused on to the moving mark and the scale readings noted. At any subsequent measurements at different temperatures and loads, increases in the length of the fibre can be measured by changes in scale readings on subsequent settings of the optical device. Calibration for initial fibre lengths l_1 at any temperatures is possible using a pure platinum fibre (say T.C., wire of 0.5 mm) of similar dimensions and with fused spherical ball ends. The length of platinum fibre can be measured accurately and its coefficient of expansion is also known. At any temperature a reading on the moving marker with the platinum fibre in position in place of the glass fibre will correspond to a known length of the fibre.

A-4.5.3 *Use of Linear Variable Differential Transformers (LVDT)*

Instead of an optical device for the measurement of δl, an extension rod from the loading device can be coupled to an LVDT (linear variable differential transformer) comprising a core and coil separated from each other. The relative movement between the core and the coil measures the displacement. This displacement is displayed or recorded as an electrical output. The possibility of adjustable amplifications for the signal is an added advantage enabling a wide range of sensitivities without disturbing the mechanical settings. LVDT's with the required sensitivities and accuracies are easily available.

A-4.5.4 Blast flame burner, two rods of stainless steel, ceramics or hard glass for flame working.

A-4.5.5 Electrically heated crucible glass melting furnace with arrangement for fibre drawing, such as water coaled copper coils; pull rod of platinum, or stainless steel of fused silica.

A-4.5.6 *Timer Devices*

Timer devices are necessary covering a range of 20 s to 2 000 s with a least count of 0.1 s. The accuracy of the time should be ensured to be better than 0.2 percent.

A-4.5.6.1 *Fibre diameter*

A micrometer screw gauge with a least count of at least 0.005 mm is required.

A-5 SAMPLE PREPARATION

A-5.1 Preparation of Fibre by Flame Working

This method can be used only when the glass is stable with heat treatment without any tendency for devitrification or phase separation. A piece of the glass about 2 to 3 cm^3 in volume is fused in the flame by holding it between the rods (of platinum, ceramics or harder glasses) and turning it around. When it is sufficiently soft it is suddenly withdrawn from the flame and drawn apart using the rods. The operation shall be completed quickly to prevent loss of volatiles in the flame working. Skill is required in this operation with respect to:

a) choice of temperature (as judged by the softness of the work pieces) at which the drawing apart is undertaken,
b) force and speed of drawing to ensure fibre of particular diameter,
c) uniformity of drawing, and
d) Stopping of drawing at the right time.

A-5.2 Preparation of Fibre from the Melt

About 100 g of the glass is melted in a platinum crucible in the electrically heated furnace. The glass can be fed in the form of cullet but the grains should not be finer than 3 mm to avoid bubbles. When the glass is completely melted and is free from bubbles a pull rod of platinum, ceramic or fused silica is dipped into the melt. The rod is pulled up quickly through a water cooled copper coil to the desired length. A similar degree of skill in the drawing is required as indicated for flame working (**A-5.1**). A mechanised device for pulling ensures repeatable and well controlled drawing speeds.

A-5.3 Specimen Preparation for Elongation Experiment

About 50 to 100 mm length of the fibre will be required for the elongation measurement. A length of about 20 to 30 mm longer than the desired length of fibre is taken and the portion of uniform round diameter is chosen. By flame working the two ends are made into spherical balls of about 2 mm. The balls are properly centred on the fibre axis.

A-5.3.1 After the fibre has cooled completely the length of the fibre is measured between the two balls, accurately to 0.2 mm. The diameter can be between 0.6 to 0.8 mm for most measurements. At the high temperature (low viscosities of 10^8 to 10^9 dPa.s) the diameter could better be up to 1 mm. A number of measurements of the diameter are made equally distributed along the length of the fibre, and at each point in two directions at right angles to each other. The maximum and minimum diameters shall not differ by more than 2.5 percent of the mean diameter. The arithmetic mean value of the diameter shall be used for calculating the fibre volume V.

A-5.3.2 The upper end of the fibre (when suspended for elongation measurements) shall be chosen to have the larger diameter ball.

A-5.3.3 The test specimen shall be free from scratches, striae, devitrification, bubbles or other inhomogeneities. There shall be no neck formation of fibre at either end close to the balls.

A-5.4 Etching of the Test Specimen

An etching solution shall be prepared with 2 mol/litre aqueous solution of hydrochloric, sulphuric or nitric acid containing 3 percent of hydrofluoric acid. The glass fibre is placed in this solution at room temperature for 0.5 to 1 min. The fibre is taken out, rinsed with distilled water and dried.

A-6 MEASUREMENT PROCEDURE

A-6.1 Calibration

The fibre elongation method based on equation (1) is an absolute method for determining the viscosity calibration involved only for thermocouples. The thermocouples must be calibrated against certified standard thermocouples at regular intervals.

A-6.2 Determination of Viscosity at Constant Temperatures

Heat the furnace to the lowest point in the temperature range within which the measurement is desired. When the furnace temperature is stable introduce the fibre with the support and extension rods. Wait for a few minutes for the temperature equilibrium to be re-established. Focus optical instrument on the moving marker and note the scale reading. In case LVDT is used, zero the chart on recorder at this point or note the reading if visual indicator only is used.

A-6.2.1 The length l_1 of the fibre in this position is known (**A-4.5.2**).

A-6.2.2 Load pieces may be added. The cathetometer/travelling microscope is focused on the mark and the timer is started. In the case of LVDT the recorder is zeroed and the chart is started simultaneously. If visual recording of LVDT output is used, a timer is started simultaneously with zeroing of LVDT output. The vernier of travelling microscope/cathetometer is moved by a known increment of length and the time when the mark crosses the reference line in the eye piece is recorded. The scale is then shifted to the next selected increment. At least six positions of lengths and the corresponding times are recorded using travelling microscope/cathetometer or LVDT for a given temperature.

A-6.2.3 Remove the loads and heat the furnace to the next higher temperature point. After stabilisation of temperature add the load pieces and repeat measurements.

A-6.2.4 At high temperatures corresponding to viscosities 10^8 to 10^9 dPa.s, no load pieces will be required. The dead load is sufficient for the measurement.

A-6.2.5 Smaller elongations with higher sensitivities for measurement of δl are preferred and the elongation during one test shall not exceed 2 percent of the starting length.

A-6.2.6 At high viscosities 10^{11} dPa.s and above delayed elastic effects become important and sufficient time shall be allowed to elapse after the addition of the load, before the readings are taken.

A-6.3 Finishing of Test Procedure

The test shall be completed before any one fibre elongates by 10 percent of l_1. Remove the test fibre from the furnace and allow it to cool to room temperature. Inspect the fibre for devitrification, striae, scratches and neckings. Again determine the length of the fibre as well as the mean diameter and note if the cross-section has deviated from circular geometry. The difference between the maximum and minimum diameter shall not exceed 4 percent of the mean.

A-7 EXPRESSION OF RESULTS

A-7.1 Corrections for Load

When the loading mass is 4 g or less, correction must be made to include the mass of the fibre and surface tension shall also be accounted for. These are taken care of by substituting for m_1 and m_2 given by:

$$m_2 = m_1 + f\rho - f\sigma$$

where

$$f\rho = 0.000\,5 \times \rho \times V$$

$$f\sigma = 0.000\,18 \times \sigma \times \sqrt{V/l_1}$$

ρ = density of the glass, in g/cc; and

σ = surface tension of the glass, in millinewton/metre.

When σ is unknown, an idea of its effect can be had using the value σ = 330 mN/m.

A-7.2 Evaluation of Measurements

A-7.2.1 A plot of length of fibre against time can be made from which, $\frac{dl}{dt}$ at different l can be evaluated. A plot of against l^2 must yield a straight line plot [*see* equation (1), **A-3.1**] from the slope of which η can be calculated. At least six points shall be plotted for straight line.

A-7.2.2 The expression given in equation (3), **A-3.2** can also be used with δl evaluated for a given l_1 and interval of time Δt. At least six different readings of δl shall be used to get average value for η.

A-7.2.3 *Representation of Results*

The viscosity values at the different temperatures may be plotted with log η on the ordinate and temperature on the abscissa. This relationship between η and T is governed by the Vogel Fulcher Tammar (VFT) equation (*see* Annex D)

$$\log \eta = A + \frac{B}{T - C}$$

This enables interpolation of log η/temperature values at points other than those at which test were done. The constants A, B, C may be determined from any three sets of η, T values from the log η, T curves.

A-8 TEST REPORT

A-8.1 The test report shall include:

a) Reference to this Indian Standard,
b) Description of sample,
c) Method of sampling,
d) Number of test specimens,
e) Method of preparation of specimen,
f) Corrections applied,
g) Plot of log versus reciprocal of temperature according to VFT equation, and
h) Any change in glass, observed during and/or after the test.

ANNEX B

(*Clause* 1)

BEAM BENDING VISCOMETER METHOD

B-1 SIGNIFICANCE AND USE

This test method is quite suitable for the measurement of viscosity of glasses in the ranges that are higher than the ones which are covered by rotational viscometry and parallel plate methods. The test method is quite useful while providing an information that is related to the glass behavior.

B-2 APPARATUS

B-2.1 The apparatus shall comprise of a furnace, a mean for controlling its heating rate and temperature, loading rod and specimen holders, and a means in order to observe the rate of viscous deflection of glass specimen.

B-2.2 Furnace

It should electrically heated by means of resistance elements. The details and the dimensions of the construction of the furnace are not critical, its cross-section can either be square with sides of 75 mm or circular with 75 mm (3 in.) diameter. The furnace should be able to maintain a constant temperature zone which covers the geometry of a specimen, including the deflection range. Differences in the temperature of the constant temperature zone are unacceptable if it is greater than 2 °C.

B-2.3 Temperature Measuring and Indicating Instruments

In order to measure the temperature, a calibrated Type K, R, or S thermocouple shall be provided. A double bore alumina tube shall be used to house the thermocouple with its junction kept within 5 mm of the specimen near to the furnace axis. The thermocouple should be referenced to 0 °C by using an ice bath, and its emf should be calculated from calibrated potentiometer that can be read with an accuracy of 60.5 °C and a sensitivity of 0.1 °C. Precautions should be taken in order to ensure that the temperature of an ice bath is maintained at 0°C during the entire test. Alternately, the value of an output given by the thermocouple can be measured on a meter which is calibrated and can be read directly. The meter (electronic thermometer) should give the value that can be read with an accuracy of 60.5 °C and a sensitivity of 0.1 °C.

B-2.4 Furnace Control

In order to maintain the temperature of the furnace at a fixed control point and for controlling the cooling and heating rates, suitable means shall be provided. Programming equipment that are commercially available can provide excellent control. A variable transformer having a manual control is quite inexpensive, but for accomplishing the required amount of control, it is quite a less adequate means.

B-2.5 Specimen Holder and Loading Rod

B-2.5.1 A support stand and a loading rod made of ceramic shall be provided so as to support the specimen and for applying the load to it. The characteristics of both the members that are related to thermal expansion must be very similar so as to decrease the motion of the loading rod that is due to expansion differences. A rectangular shaped muffle of alumina or circular shaped tube that can be notched to define the position of the specimen can be a suitable support stand (*see* Note). The surfaces of these notches that are supporting shall be flat and thus lies in a plane that is perpendicular to the furnace axis. The inner edges of notches define the span of the support once the beam of the specimen starts deflecting. A span of support being around 5 cm (2 in.) is recommended. An appropriate loading rod can be provided by a single-crystal sapphire rod flame which can be bent at one end into a shepherd's crook form. The crook shall be able to make a contribution to the load on the specimen, thus its weight should be kept to a minimal value.

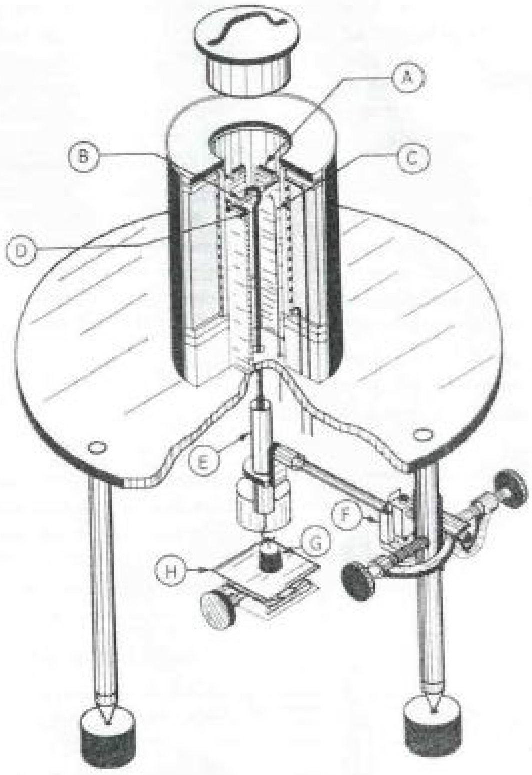
NOTE — One of the most suitable material for both support stand and loading rod is vitreous silica. Thus, it is not suggested for temperatures higher than 900 °C.

B-2.6 Extensometer for Measuring Midpoint Deflection

The method for observing the deflection rate of the specimen shall permit an appropriate reading of total deflection of minimum 10 mm. Direct reading of 0.010 mm and estimates of 0.001 0 mm shall be allowed by the specimen. It should have such an accuracy that the indication error will not exceed 62 percent for any deflection value measured. This will limit the minimum value of deflection so that it can be used in the calculation. For this purpose, a linearly variable differential transformer (LVDT) is suitable just like is any other device (for example, optical or capacitive), provided that as specified, deflection is reliably measured.

B-2.7 Weights

Weighing sets spanning the range from 1 to 500 g and with an accuracy of 0.1 percent relative is required.



A – Alumina muffle support stand
B – Specimen Beam
C – Thermocouple
D – Loading rod
E – Linearly variable differential transformer (LVDT)
F – Zero-adjust mechanism for LDT
G – Weight
H – Laboratory jack

FIG. 4 CUTAWAY DRAWING OF BEAM-BENDING APPARATUS

B-2.8 Micrometre Calipers

Micrometre calipers that are readable to an accuracy of at least 0.01 mm are needed in order to measure the dimensions of the specimen.

B-2.9 Analytical Balance

An analytical balance that is capable of weighing the loading train and shepherd’s crook to an accuracy of 0.1 percent relative.

B-3 PREPARATION OF TEST SPECIMEN

B-3.1 Specimens may either be drawn by flame or center-less ground into cylindrical form or mill ground and diamond-saw cut into rectangular form. Non-uniformity in any dimension that is present along the length of the specimen shall not exceed by 2 percent. An average value should be used in case the non-uniformity in case of dimension exists.

B-3.2 The ratio of the beam span to moment of inertia shall not be less than 60. The thickness or diameter to span ratio shall be less than 0.1.

B-4 CALIBRATION

B-4.1 Make a direct calibration of the apparatus by using standard glasses, having known temperature values over the particular range of viscosity covered by this practice. Any bias should be corrected by an overall instrument calibration.

B-4.1.1 Find the viscosity by the use of test beams of SRM glass that covers a range of cross-sectional moments of inertia. Make the viscosity measurement over the viscosity range of 10^8 Pa.s to 10^{11} Pa.s by making use of the standard procedure given in **B-5**. Perform the tests by keeping the span and time-temperature function constant.

B-4.1.2 Fit resulting data mathematically in a convenient form (for example, polynomial or Fulcher equation). After that fit the data supplied for the glass SRM in a Fulcher equation.

B-4.1.3 From both equations, calculate the viscosities that are determined in **B-4.1.2** at 20 °C with the minimum intervals over the range of measurement. Find out the viscosity ratio $\eta_{SRM.fit}/\eta_{measured.fit}$ = fractional correction, and the draw a fractional correction versus log viscosity (measured fit) calibration curve. This can be used for the correction of the experimental viscosity data (*see* Note). Corrections that are greater than 20 percent are the cause of concern and thus initiation of apparatus troubleshooting should be done.

NOTE — In case under some heating or cooling rate time-temperature function, analyses are performed, the temperature of the thermocouple may lag or lead the actual temperature of the sample. In case lag or lead in the thermocouple occurs, the calibration curve described in **B-4.1.3** would include the given temperature and viscosity bias. In order to find out if thermocouple lag or lead exists, measure the viscosities for a glass SRM keeping isothermal conditions at several temperatures. Make a comparison of temperatures at equivalent viscosity levels by analyzing the same glass SRM which is measured under the cooling or heating rate condition. Lag or lead in the thermocouple can be indicated from the differences in the temperature. This difference should be applied as a correction in the measured value of temperatures before drawing the calibration curve (*see* **B-4.1.3**) or applying the calibration correction to the data tested.

B-5 PROCEDURE

B-5.1 Take the deflection data under isothermal conditions or during cooling or heating at controlled rates so that it does not exceed 5 °C/min.

B-5.2 Identify the function of time-temperature (for example, 5 °C/min heating rate) which is to be used in the test. Use an alumina or sapphire specimen so as to generate a background deflection against temperature curve, by making a use of the chosen time-temperature function intended for the measurement of the specimen. The deflection that took place in the test specimen is measured by the algebraic subtraction of the background curve from the curve obtained from measured values.

B-5.3 Make the measurement of the dimensions of the test beam to the nearest 0.01 mm. The data is then used to calculate the cross-sectional moment of inertia.

B-5.4 Place a thin platinum foil in each notch and after that place the specimen beam across the support stand at notch points so as to protect the support from reacting with the specimen and reducing the friction between the specimen and support. Place a thin foil of platinum in between the specimen and the loading rod. All the foils of platinum must be of similar thickness, and are suitably thin (preferably 25 µm thick) so that it can allow the seating of the components in their appropriate position.

B-5.5 Engage the loading rod to the specimen carefully and center it. Now on the end of the extensometer, apply a weight to the hook by adjusting the total, applied load (that consists of the specimen, hooks, loading rod fixtures, and weight) such that a usable rate of deflection is obtained. Adjust the extensometer position to the lower end of its measuring range. Start the heating of the furnace, by making the use of the time-temperature function that is chosen for measurements.

B-5.6 When a usable rate of deflection is achieved, begin the recording of extensometer, time, and temperature data. This data can be used for data reduction. The time duration for the collection interval should not exceed more than 1 min. Suitable ways of accumulating the data include computer controlled data acquisition or by plotting the deflection and temperature of the specimen by using a two pen recorder that operates on a convenient time base. (If such kind of recording device is not available then data should be manually taken, the temperature and deflection may be recorded by taking the readings of both the temperature and extensometer alternately at an interval of 30 s such that each one is read at 1 min intervals. As it has less accuracy than the other available methods, the user is generally discouraged from using this method in order to acquire data). In case the extensometer goes off range while performing the test, it is desirable to reset it. Total beam deflections that are greater than 10 mm are excessive.

B-6 CALCULATION

Make the use of the changes (that are corrected) in extensometer readings, *dh*, in the time interval that has been given, *dt*, as the rate of deflection of the midpoint, *dh*/*dt*, at the temperature that corresponds to the middle of that interval. Substitute these data points into equation (1) so as to calculate the viscosity, *h*. Correct the viscosity with the help of the calibration curve by multiplying it with the fractional correction factor that corresponds to that viscosity.

B-7 TEST REPORT

The test report shall include:

a) Reference to this Indian Standard;
b) Identification of the glass piece that is tested;
c) Source of manufacture and date;
d) Reference for calibration;
e) Viscosity points and temperature;
f) Date of the test; and
g) Other observations (for example, sample that is crystallized during measurement).

B-8 PRECISION AND BIAS

B-8.1 Precision

In case the round robin testing is absent, it is not possible to make a particular precision statement. It is possible to estimate a precision from data scatter in mathematical curve fitting.

B-8.2 Bias

The given procedure should yield viscosity data to 6-10 percent of referenced SRM values. Systematic departures can be seen for the values that are obtained near the starting and at the end of the determination, the points where the respective rates of deflection are extremely small or large. A rigid apparatus test is done by caliberating with one NIST SRM glass and then make the measurement for another NIST SRM glasses based on the calibration. In case the values of the other standard glasses are within 6-10 percent of certification, satisfactory performance is established. In case the errors that arises increases or decreases with viscosity, a problem in the measurement of temperature may exist or large thermal gradients may be observed in the furnace. These should be corrected.

ANNEX C

(*Clause* 1)

VISCOUS COMPRESSION OF A SOLID RIGHT CYLINDER

C-1 SIGNIFICANCE AND USE

This test method is suitable for measuring the viscosity of glasses in between the range under which rotational viscometry is beneficial and the range under which beam bending viscometry is beneficial. This is useful in order to determine the viscosity/temperature curve in the region which is near to the point of softening. The given test method is beneficial in giving the information which relates to the glass behavior.

C-2 APPARATUS

The apparatus comprises of a furnace as a means of controlling and measuring its heating rate and temperature, loading rod and specimen holders, and a means so as to measure the rate of viscous compression of the glass specimen.

C-2.1 Furnace

The furnace should be heated electrically by resistance elements. The details and the dimensions of the construction of furnace are not critical; it can have a circular cross section of 75 mm (≈ 3 in.) diameter or can also have square of sides of 75 mm. The temperature zone of the furnace shall be constant that it covers the specimen geometry, including the compression range. Differences in temperature of greater than 2 °C within the constant temperature zone are unacceptable.

C-2.2 Temperature Measuring and Indicating Instruments

In order to measure the temperature, a calibrated Type K, R, or S thermocouple shall be provided. A double bore alumina tube shall be used to house the thermocouple with its junction kept within 5 mm of the specimen near to the furnace axis. It is desirable that the thermocouple should be referenced to 0°C by using an ice bath, and its emf should be calculated from calibrated potentiometer that can be read with an accuracy of 60.5°C and a sensitivity of 60.1°C. Precautions need to be taken in order to ensure that the ice bath is maintained at 0°C during the test. Alternatively, the output value from the thermocouple can be measured on a direct reading meter (electronic thermometer) which is calibrated and can be read with ± 0.1°C sensitivity and ± 0.5°C accuracy.

See Note below **B-4.1.3** for temperature lag-lead corrections.

C-2.3 Furnace Control

In order to maintain the temperature of the furnace at a fixed control point and for controlling the cooling and heating rates, suitable means shall be provided. Programming equipment that are available commercially provides excellent control. A variable transformer having a manual control is inexpensive, but is relatively less adequate means to accomplish the required control.

C-2.4 Specimen Holder and Loading Rod

A typical type of configuration is shown Fig. 1. On top of the support stand is a stationary plate (*see* Note) positioned, which is 6 mm (1/4 in.) thick by diameter of the support stand. A movable plate which is 6 mm thick by 44 mm minimum diameter and is parallel and concentrically placed above the fixed plate (*see* parallel plates in Fig. 5). To the center of the movable plate at the top, in a shrink fit configuration is a 9 mm (3/8 in.) diameter ground alumina rod which is center less and is of length which is sufficient to reach approximately 150 mm (6 in) beyond the furnace top or its supporting

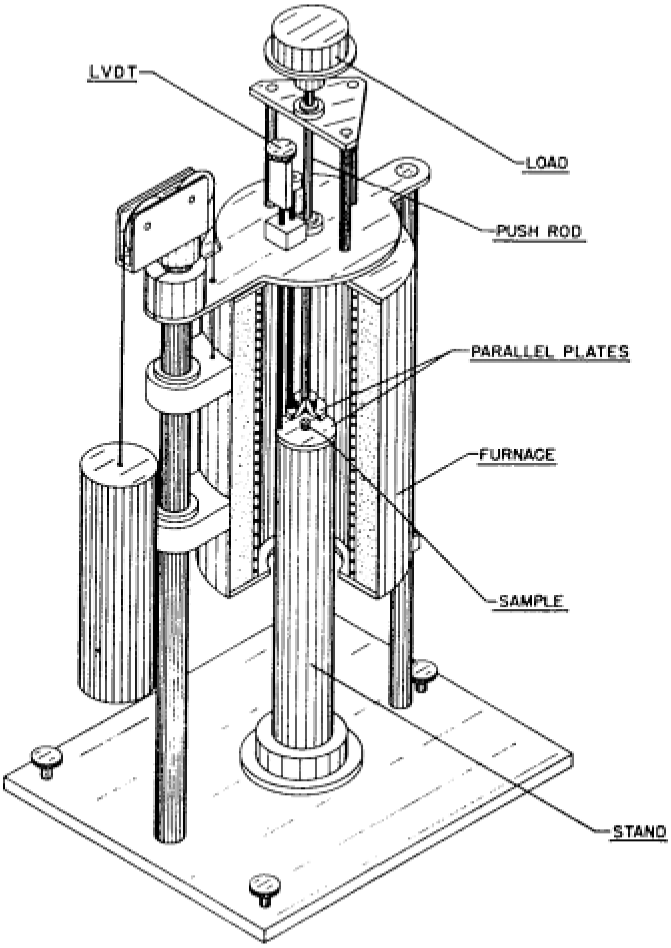


FIG. 5 A TYPICAL PARALLEL PLATE VISCOMETER

structure, or both. The assembly thus provides a mean so as to load a specimen while measurement. Bushings that are attached to the outer frame guide the shaft with a minimum amount of friction.

NOTE — Suitable materials for the assembly components are vitreous alumina and silica, as they are noble or low expansion metals which used in pairs. The temperature limitations for these alternate materials must be observed by the user.

C-2.5 Extensometer for Measuring Thickness Change

The way of observing the rate of change in thickness of the specimen should allow the reading of total change of at least 6 mm. The direct reading of 0.010 mm shall be allowed by the extensometer with an estimate of 0.001 mm. The accuracy shall be such that the indication error shall not exceed 62 percent for any translation being measured. This will limit the rate of translation such that it may be used in calculation. For this purpose, a linearly variable differential transformer (LVDT) is suitable just like any other device (for example, optical or capacitive) with a condition that the changes in the length measured reliably as specified.

C-2.6 Micrometer Calipers

Micrometer calipers, readable to an accuracy of at least 0.01 mm are needed in order to measure the dimensions of the dimensions.

C-3 PREPARATION OF TEST SPECIMEN

Specimens that are needed for this test method are right, circular cylinders and small. Generally, the dimensions are 6 mm (1/4 in.) to 12 mm (1/2 in.) diameter and are 3 to 6 mm thick. Specimens can either be sliced from a rod or can be core-drilled from flat stock. In both cases, surfaces that are flat must be polished and ground to be plane-parallel to 60.001 mm. Cylinders that are made at high pressure by dry pressing of frit can provide meaningful data.

C-4 CALIBRATION

C-4.1 Calibration of the apparatus can be done directly by using standard glasses, having known temperature and viscosity values. Bias is required to be corrected by overall calibration of an instrument.

C-4.1.1 Find the viscosity by using the test cylinders of calibrating glasses that cover a wide cross-sections range expected for use in routine testing. Procedure given in **C-5** and **C-6** shall be used to measure the viscosity.

C-4.1.2 Fit resulting data mathematically to a convenient form.

C-4.1.3 By using both the equations, calculate the viscosity as determined in **C-4.1.2** at 20 °C with the minimum intervals over the range that is measured. Find the viscosity ratio, $\eta_{SRMfit}/\eta_{measured\ fit}$ = fractional correction, and plot a calibration curve between fractional correction versus log viscosity (measured fit). This can be used to correct the experimental data for viscosity (*see* Note). Corrections which are greater than 20 percent are the reason for concern and therefore the troubleshooting of the apparatus is recommended.

NOTE — In case the analysis are performed under some cooling or heating rate time-temperature function, the temperature of the thermocouple may lag or lead the actual temperature of the sample. In case the lag or lead in the thermocouple does not occur, the calibration curve being described in **C-4.1.3** would include this temperature and viscosity bias. In order to find if thermocouple lag or lead exists, it is recommended that the viscosities for a glass SRM are measured under isothermal conditions at several temperatures. Make a comparison of the temperatures at equivalent viscosity levels from the analysis of the same glass SRM being measured under the cooling or heating rate condition. The differences in the temperature indicate the lag or lead in the thermocouple. The difference should be applied to the correction in the temperature being measured prior to generating the calibration curve (*see* **C-4.1.3**) or applying the calibration correction to the data being tested.

C-5 PROCEDURE

C-5.1 Take the deflection data under isothermal conditions or during cooling or by heating at controlled rates so that it does not exceed 5 °C/min.

C-5.2 Identify the function of time-temperature (for instance, heating rate of 5 °C/min) that is to be used in the test. Plot a curve of background deflection against temperature by measuring with the upper loading plate which is in contact with the lower support plate (pieces of platinum foil may be used for separation) during the operation of the furnace under time-temperature function being chosen. The change in thickness of the test specimen is determined by making the algebraic subtraction of the curve at the background from curve value being measured.

C-5.3 Measure the diameter of the specimen and its thickness by using a micrometer to within 0.01 mm and carefully record the obtained results.

C-5.4 In order to protect the parallel plates from being reacted with the specimen, keep the specimen between the pieces of two platinum foil and place the sandwiched specimen in between the parallel plates concentrically. All the foils of platinum must be of similar thickness, and are suitably thin (preferably 25 µm thick) so that it can allow the seating of the components in their appropriate position.

C-5.5 Make an adjustment of the position of an extensometer to the lower end of its measuring range. Keep the furnace at its position and then initiate the chosen time-temperature function.

C-5.6 When a deflection rate that is usable is attained, begin the recording of extensometer, temperature and time data that can be used in reduction of data. The interval of collection should not increase by more than 1 min. Suitable means in order to gather the data include computer controlled data acquisition or making the plot of the change in thickness and temperature of the specimen by using a two pen recorder that operates on a convenient time base. (If such kind of recording device is not available then data should be manually taken, the temperature and change in thickness may be recorded by taking the readings of both the temperature and extensometer alternatively at an interval of 30 s such that each one is read at an interval of 1 min. As it has less accuracy than the other available methods, the user is generally discouraged from using this method in order to acquire data. In case the extensometer goes off range while performing the test, it is desirable to reset it. Compression of the sample to a thickness less than 1 mm is excessive.

C-6 CALCULATION

Make the use of the changes (that are corrected) in extensometer readings, *dh*, in the time interval that has been given, *dt*, as the rate of change of thickness, *dh/dt*, at the temperature that corresponds to the middle of that interval. Also measure the change in thickness of the specimen, *d*, at the middle of the time interval; making its use to calculate the thickness of the specimen.

$$h = h_0 - \mathrm{d}$$

where

h_0 = the initial specimen thickness.

Substitute the data in the equation (1) in order to calculate the viscosity, η. Correct the viscosity with the help of the calibration curve (*see* **C-4**) by multiplying it with the fractional correction factor that corresponds to that viscosity.

C-7 TEST REPORT

The test report shall include:

a) Reference to this Indian Standard,
b) Identification of the glass that has been tested,
c) Source and date of manufacturing,
d) Reference of calibration,
e) Viscosity and temperature points,
f) Date of the test, and
g) Other observations like sample crystallized during measurement.

C-8 PRECISION AND BIAS

C-8.1 Precision

In case the round robin testing is absent, it is not possible to make a particular precision statement. It is possible to estimate the precision from the data scatter in mathematical curve fitting of data.

C-8.2 Bias

The given procedure should yield the values of the viscosity points to ± 10 percent of referenced SRM values. It is possible that systematic departures may occur in case of the values which are obtained near the start and towards the end of the determination where the rate of change of thickness is small. A rigid test for an apparatus is the one that requires the calibration with NIST SRM glass and then measure other NIST SRM glasses based on the calibration. In case the other standard values of glass are within 4 °C of certification, satisfactory performance can be established. If errors that increases or decreases with viscosity arises, a problem due to measurement of temperature may exist or there is a possibility that the thermal gradients in the furnace are too large. These should be corrected.

ANNEX D

(*Clauses* A-1 *and* A-7.2.3)

VISCOSITY - TEMPERATURE RELATIONSHIP

D-1 THE VFT-EQUATION

D-1.1 For the purpose of interpolation the viscosity/temperature relationship is conveniently described by the equation of Vogel Fulcher and Tammann (the VFT-equation):

$$\log \eta = A + \frac{B}{T - C} \qquad \dots (1)$$

The value of the dynamic viscosity η_{neg} shall be inserted in decipascal per second (dPa.s), and the temperature T in degree celsius (°C).

The constants *A, B* and *C* are characteristics for the glass under test.

NOTE — The order of magnitude of the constants for technical glasses is as follows:

A = 10 (decadic logarithm of viscosity in decipascal second);
B = 10^3°C; and
C = 10^2°C.

D-2 DETERMINATION OF THE CONSTANTS

Three pairs of measured temperatures and equilibrium viscosities is equal to l, 2, 3, covering the range of the measurement and separated sufficiently from each other, are chosen to calculate the constants of the VFT-equation by means of equations (2) to (4).

D-3 TEMPERATURE COEFFICIENT OF VISCOSITY

The temperature coefficient of the viscosity of a glass is defined by equation (5):

$$U_n = \frac{1}{\eta} \times \frac{d\eta}{dt} \qquad \dots (5)$$

Using the VFT-equation, the temperature coefficient U_n is given by equation (6):

$$U_n = \frac{2.303 \times B}{(T - C)^2} \qquad \dots (6)$$

D-4 ERROR CHARACTERIZATION

The deviation of a measured point from a fitted temperature/viscosity curve (for example found by regression analysis) can be expressed either by a viscosity difference Δlog η or by a temperature difference Δ*T*. These differences are related to one another by equation (7):

$$\Delta \log \eta = 0.434\,3 \times U_n \times \Delta T \qquad \dots (7)$$

Where U_n is the mean temperature coefficient of viscosity in the corresponding range of temperature.

D-5 VISCOSITY/TEMPERATURE PLOT

The graphical representation of the viscosity/temperature relationship is usually performed by plotting the logarithm of viscosity as ordinate against the linear temperature axis as abscissa.

$$C = \frac{(T_2 - T_1)(T_a - T_1) \times (log\ \eta_a - log\eta_2)}{\begin{gathered}(T_2 - T_1)(log\ \eta_a - log\eta_1)\ (T_2 - T_1)\\ (log\ \eta_2 - log\ \eta_1)\end{gathered}} + T_1 \qquad \text{... (2)}$$

$$A = \frac{log\ \eta_2 (T_2 - C) - log\ \eta_1 \times (T_1 - C)}{(T_2 - T_1)} \qquad \text{… (3)}$$

$$B = (T_1 - C) \times (log\ \eta_1 - A) \qquad \text{… (4)}$$

As a proof of the evaluation, calculate the constant B once more, using equilibrium viscosity is equal to 2 or 3, respectively.

ANNEX E

(*Foreword*)

DEPARTURE FROM ISO DOCUMENT

E-1 The departure from ISO document is as follows.

E-2 ELONGATION RATE

While describing the procedure for determination of elongation of the fibre, the ISO document prescribes that after loading the fibre, one should wait 'for nearly constant elongation rate', before proceeding to record elongation as a function of time. It is apparent from the basic expression relating elongation rate to at a given temperature that the elongation rate is directly proportional to l^2. Hence elongation rate will never be constant. This has been taken into account.

E-3 INITIAL LENGTH OF FIBRE

A cathetometer or telemicroscope focussed on a well-defined mark on the extension rod or loading device for the fibre enables measurement of increments in length of the fibre. However, the initial length of the fibre inside the furnace at the test temperature must be known for correlating the elongation rate to the viscosity at the temperature. This is normally taken care of by measuring the length of fibre at room temperature using vernier calipers or travelling microscopes. The measuring device is focussed on the mark after suspending the fibre at room temperature inside the furnace. As the furnace temperature is raised, increments in length are monitored by following the moving mark on the optical measuring device. However, this procedure makes it imperative that the optical measuring instrument must be absolutely stationary with rigid fixing devices throughout the measurement procedure for the entire range of temperatures and hence the entire measurement must be completed in a day.

The use of a pure platinum wire (T. C. grade) of about 0.5 mm diameter or more will provide a very convenient standard length with reference to which positions of initial readings of optical measuring devices can be related to specific initial lengths in the furnace. The platinum wire must have spherical ends by flame fusion, in an oxy-acetylene flame or electric arc and suspended inside the furnace at any desired temperature in the same manner as the glass fibre. The coefficient of linear thermal expansion of pure platinum (T. C. grade) is well defined and thus an accurate initial length position can be known corresponding to the reading of the optical devices on the standard mark when the platinum wire is suspended at any temperature and even after an interruption in the procedure entailing disturbance of the optical measuring devices. The use of the platinum wire has, therefore, been incorporated.

E-4 MEASUREMENT PROCEDURE

E-4.1 Recording of increment in length as a function of time at any given temperature enables a plot of *dl*/*dt versus* l^2. This must be a straight line yielding an elegant method for calculating η from the slope. This procedure has been incorporated in preference to calculating η from individual values of l_1, l_2 and the corresponding time interval.

E-4.2 The dynamic methods for measurement involving determination of elongation rates while the furnace temperature is programmed for linear rise and fall has not been included since it is felt that the precision of rate control and exact repeatability of programmes may be very difficult to achieve.

ANNEX F

(*Foreword*)

COMMITTEE COMPOSITION

Glass, Glassware and Laboratoryware Sectional Committee, CHD 10

Organization	*Representative(s)*
CSIR-Central Glass and Ceramic Research Institute, Kolkata	Dr Suman Kumari Mishra (***Chairman***)
AGI Glaspac, Gurugram	Shri Rajesh Khosla Shri Vishwanath Ambadipudi (*Alternate*)
Asahi India Glass Ltd, New Delhi	Shri Nagendra Kumar Shri Navin Rai (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	Dr (Shrimati) Madhumita Goswami Dr P. N. Nandi (*Alternate*)
Borosil Ltd, Mumbai	Shri Shrikant Gangan Shri Satish Chitriv (*Alternate*)
Building Materials and Technology Promotion Council, New Delhi	Shri A. K. Tiwari Dr Amit Rai (*Alternate*)
Central Building Research Institute, Roorkee	Dr Navjeev Saxena Shri Ajay Chaurasia (*Alternate*)
CSIR-Central Glass and Ceramic Research Institute, Kolkata	Dr K. Annapurna Shri Sitendu Mandal (*Alternate*)
Centre for the Development of Glass Industry, Firozabad	Shri Sanjeev Chinmalli Shri Devendra Sah (*Alternate*)
Confederation of Construction Products and Services (CCPS), New Delhi	Shri Deepak Gahlowt Shrimati Sarita Balodhi (*Alternate*)
Controllerate of Quality Assurance (Materials), New Delhi	Shri Sunil Kumar Shri Brijesh Singh Tomar (*Alternate*)
Department for Promotion of Industry and Internal Trade, New Delhi	Shri T. S. G. Narayannen Shri Uday Singh Mina (*Alternate*)
Federation of Safety Glass, Faridabad	Shri Sharanjit Singh Shri Gurmeet Singh (*Alternate*)
Glazing Society of India, New Delhi	Shri G. N. Gohul Deepak
Gold Plus Glass Industry Ltd, New Delhi	Shri Vivek Dubey Shri Prem Dutt (*Alternate*)
Govt College of Engineering and Ceramic Technology, Kolkata	Dr Rituparno Sen Dr (Ms) Kaberi Das (*Alternate*)
Hindustan Glass Works Ltd, New Delhi	Shri Varun Gupta Shri K. Arvin (*Alternate*)
Hindustan National Glass and Industries Ltd, Kolkata	Shri Pawan Bharadwaj Shri Prasanta Ash (*Alternate*)
Indian Institute of Packaging, Mumbai	Dr Tanweer Alam Shri Bidhan Das (*Alternate*)
National Test House, Kolkata	Dr S. K. Kulshrestha Shri D. V. S. Prasad (*Alternate*)
Office of the Development Commissioner (MSME), New Delhi	Shri R. K. Bharti Shri Santosh Kumar (*Alternate*)

Organization	*Representative(s)*
Saint-Gobain Glass India Ltd, Chennai	Shri A. R. Unnikrishnan Shri Chiranjit Roy (*Alternate*)
Schott Glass India Pvt Ltd, Mumbai	Shri Anand Bakshi Shri Hansraj Goud (*Alternate*)
Shriram Institute for Industrial Research, Delhi	Shri Aneesh Kumar Shri Joy Banerjee (*Alternate*)
Shree Labware and Process Systems, Bengaluru	Shri A. N. S. Kumar
SISECAM Flat Glass India Pvt Ltd, Halol	Shri Parag Shah
The All India Glass Manufacturers Federation, New Delhi	Shri Sourabh Kankar Shri Vinit Kapur (*Alternate*)
Voluntary Organization in Interest of Consumer Education (VOICE), New Delhi	Shri B. K. Mukhopadhyay Shri K. C. Chaudhary (*Alternate*)
In Personal Capacity, (*D 60/23 Chhote Gaivi Varanasi, U.P. 221010*)	Dr Devendra Kumar
BIS Directorate General	Shri A. K. Lal, Scientist 'E' and Head (CHD) [Representing Director General (*Ex-officio*)]

Member Secretary

Shri Mohit Garg
Scientist 'B' (CHD), BIS

Terminology, Code of practice, Methods of Sampling and Tests Subcommittee, CHD 10:1

Organization	*Representative(s)*
CSIR-Central Glass and Ceramic Research Institute, Kolkata	Dr K. Annapurna **(*Convener*)**
All India Glass Manufacturers Federation, New Delhi	Shri Vinit Kapur Shri Sourabh Kankar (*Alternate*)
Asahi India Glass Ltd	Shri Nagendra Kumar
Borosil Ltd	Shri Shrikant Gangan Shri M. V. Ramana (*Alternate*)
Federation of Safety Glass	Shri Farhat Kamil Shri Tariq Kachwala (*Alternate*)
Glazing Society of India	Shri G. N. Gohul Deepak
Gold Plus Glass Industry, New Delhi	Shri Vivek Dubey Shri Prem Dutt (*Alternate*)
Gujarat Guardian Ltd	Shri Sandeep Singh
Saint-Gobain Glass India Ltd	Shri Chiranjit Roy

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Head (Publication & Sales), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: CHD 10 (18010).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices: *Telephones*

Central : 601/A, Konnectus Tower-1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002 — 2323 7617

Eastern : 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091 — 2367 0012, 2320 9474

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg Chandigarh 160019 — 265 9930

Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113 — 2254 1442, 2254 1216

Western : Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093 — 2821 8093

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. CHANDIGARH. CHENNAI. COIMBATORE. DEHRADUN. DELHI. FARIDABAD. GHAZIABAD. GUWAHATI. HIMACHAL PRADESH. HUBLI. HYDERABAD. JAIPUR. JAMMU & KASHMIR. JAMSHEDPUR. KOCHI. KOLKATA. LUCKNOW. MADURAI. MUMBAI. NAGPUR. NOIDA. PANIPAT. PATNA. PUNE. RAIPUR. RAJKOT. SURAT. VISAKHAPATNAM.

Published by BIS, New Delhi